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6. AUTHORS
Jochen Fiebig, Lilia Kurmanaeva and Amiya Mukherjee

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES
University of California - Davis
1850 Research Park Drive
Suite 300
Davis, CA 95618 -6153

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14. ABSTRACT
Multilayer metallic materials have recently shown to possess high strength and thermal stability. This study focuses on the mechanical response of multilayered Cu- Nb multilayers at room and elevated temperature, which can be potentially interesting for Army applications. Tensile strength and fracture morphologies are discussed.

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Report Title

Final Report: An Investigation on the Mechanical Behavior of Roll-Bonded Multilayered Cu-Nb Nanocrystalline Materials

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Multilayer metallic materials have recently shown to possess high strength and thermal stability. This study focuses on the mechanical response of multilayered Cu- Nb multilayers at room and elevated temperature, which can be potentially interesting for Army applications. Tensile strength and fracture morphologies are discussed.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

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TOTAL:

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Received Paper

TOTAL:

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Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

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Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

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(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received	Book

TOTAL:

Received	Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

None

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
	0.00	
FTE Equivalent:	**0.00**	
Total Number:	**0**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
Jochen Fiebig	0.30
FTE Equivalent:	**0.30**
Total Number:	**1**

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Amiya Mukherjee	0.00	No
FTE Equivalent:	**0.00**	
Total Number:	**1**	

Names of Under Graduate students supported

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FTE Equivalent:	
Total Number:	

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This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

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Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

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Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME
Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

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An Investigation on the Mechanical Behavior of Roll-Bonded Multilayered Cu-Nb Nanocrystalline Materials.

Final Report:

Grant number: W911NF-13-1-0492

Jochen Fiebig, Lilia Kurmanaeva and Amiya K. Mukherjee

1. *Introduction*

Multilayered metallic materials have been in the focus of materials science in recent years due to their advanced properties like high strength, thermal stability, and ultrahigh resistance to radiation damage. This study focuses on the mechanical response of multilayered Cu/Nb at elevated temperatures which could be potentially interesting for Army applications.

In this type of material the bi-material interphases (here between the Cu and Nb) play an important role for the understanding of the observed properties, in particular if the layer thickness decreased down to less than 100 nm. Due to the high fraction of these interfaces and their properties to absorb and eliminate defects the behavior of the materials is 'interface dominated'. At a length scale above one micron the materials behavior is considered to be 'constituent dominant' as it is for conventional alloys and composites.

ARB (accumulative roll bonding) has been widely used to manufacture multilayered Cu/Nb specimens [1,2]. Previous works have demonstrated that under these extreme strains, reached in the ARB process, dominant specific interphase structures are formed [3,4]. An important goal is to understand the influence of these specific interfaces on the mechanical behavior at elevated temperatures to be able to understand and control desired materials properties .

2. *Sample preparation and experimental methods*

The samples were produced in Los Alamos Lab by accumulative roll bonding (ARB). At the beginning one Nb-sheet is sandwiched between two Cu-sheets. All three plates are rolled together. Then the rolled sheet is cut in half, both sides are brushed, stacked on top of each other are rolled again. This procedure is repeated multiple times until the starting thickness of 1-2 mm is reduced down to approximately 0.5 mm. At the end of the process the multilayered sheet had a layer thickness of 30 nm. Earlier studies have revealed that both metals do not intermix with each other. Instead they exhibit special orientation relationship between the neighboring Cu and Nb layer.

The main focus of the presented study was the mechanical properties at elevated temperatures. Tensile specimens were cut out of the Cu/Nb sheet by using EDM. The gage section of the dog-bone shaped samples had a length of 3 mm and a width of 1 mm, see fig.1. Samples were cut out parallel and perpendicular to the rolling direction.

The tensile tests were conducted with a precision mini-tensile tester controlled via a Labview-program. A load cell with a resolution of 0.01 N was used to measure the applied force while obtaining the displacement with a resolution of 5 micrometer. For a tensile test at higher

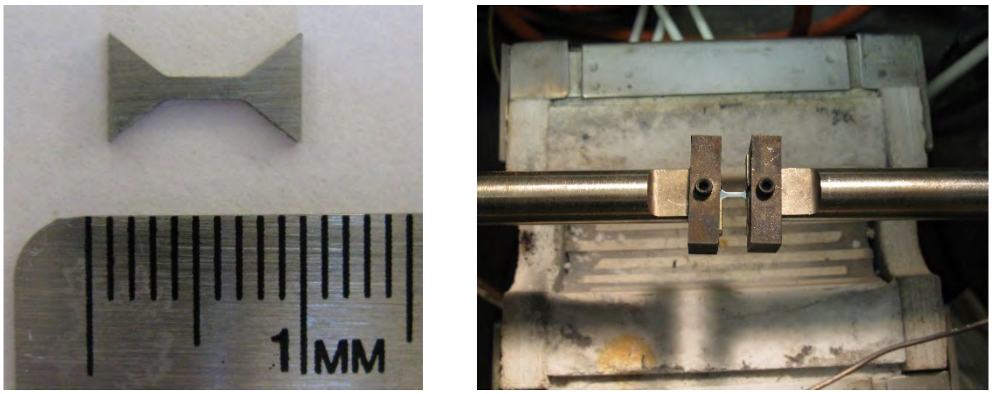

Fig. 1: left) tensile specimen after EDM cutting. The total length is 11 mm, 3 mm the gage length. right) polished sample in the grids of the mini tensile tester

temperature the chamber is first evacuated and backfilled with high-purity Argon to protect the sample from oxidation.

Prior to the tensile tests the sides of the gage section were ground with SiC paper (up to 4000) and the top and bottom surfaces of the tensile specimens were polished with standard metallography methods to a mirror-like quality. This should prevent preferred nucleation of cracks from the outer surfaces in the gaga section. The fracture surfaces was analyzed after the test by using the FEI XL30 SEM and the Scios FEM/FIB from FEI.

3. *Experimental results*

Fig 2a) shows a comparison of the stress-strain curves perpendicular and parallel to the rolling direction measured at room temperature. The elongation to failure for both samples is limited to less than 15%. The sample that was tested perpendicular to the rolling direction exhibit higher stress than the sample tested parallel to the tensile direction, 1625 MPa and 1275 MPa respectively. The difference is 350 MPa.

The fracture surface after the tests at room temperature was observed with an SEM. An overview image and some images obtain at higher magnification are presented in fig. 3 and 4.

At elevated temperature, in this case 500 C, the ductility is still limited to less than 15%. The ultimate stress drops for both tested samples, perpendicular and parallel to the tensile direction, by around 1 GPa and 800 MPa respectively.

4. Discussion

As already discussed in pervious works by Mara et al. [4] Cu/Nb multilayered samples with a layer thickness of 40 nm the limited ductility coincides well with the onset of plastic instability based on the Considere criterion. This also seems to apply for the present tensile tests performed at room temperature for Cu/Nb samples with 30 nm layer thickness (see fig.2 a). The samples fracture without any observable necking as it can be seen from the overview SEM fractographs in fig. 3a) and 4a). Furthermore one can recognize layer delamination, especially

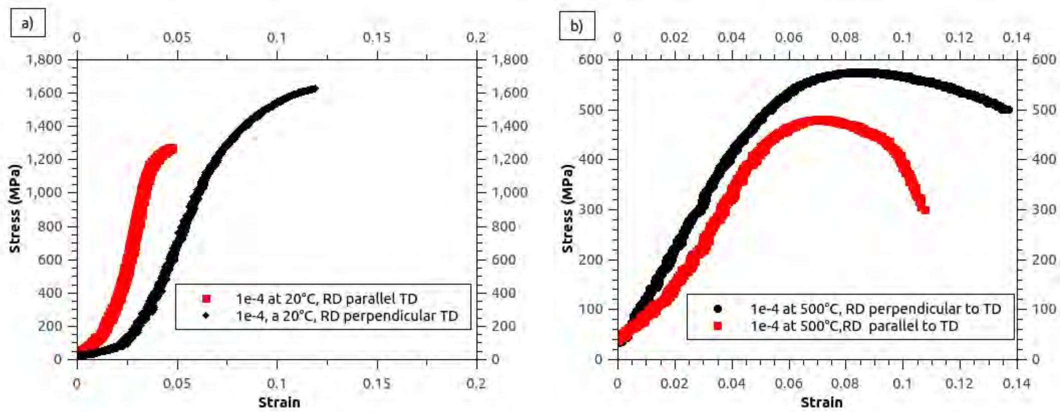

Fig. 2: a) Stress strain curves of multilayered Cu/Nb with a layer thickness of 300 nm obtained at room temperature and b) at 500 C.

close to the middle of the sample. This is not surprising since these are the last interphase interface that
got stacked together in the ARB process. It is not unexpected that the bonding between these two material surfaces is the weakest in the center of the sample.
The remarkable difference (about 350 MPa) of the fracture stress at room temperature between the tested samples perpendicular and parallel to the tensile direction might be explained by the anisotropy of the grain shape. It is known that rolling will lead to elongated grain along the tensile direction. Since grain boundaries due to the Hall-patch relationship strengthen the material, it is possible that a part of the increased stress in the samples tested perpendicular to the tensile direction is due to the higher fraction of grain boundaries.
At elevated temperature it can be clearly observed that the stress strain curves reach a maximum, the ultimate tensile stress, followed by a decrease of the stress level. This indicates plastic instability that is caused by necking of the samples. The ultimate tensile stress is lower (about 100 MPa lower) than the stress level reached in Cu/Nb multilayered samples with 40 nm layer thickness at 500 C [5] (about 700 MPa). In contrast to our result Mara et al. [5] did not observe the ultimate tensile stress since the samples fractured as soon as plastic instability starts. The tests in the literature were performed with the tensile direction oriented parallel to the rolling direction, a study about the anisotropy is still missing.
Since the fracture surface was influenced by oxidation due to a leak in the vacuum chamber (during the duration of this grant period), no reproducible fractographs could be obtained to support this observation.

5. References

[1] Lim, S.C.V. and A.D. Rollett, *Length scale effects on recrystallization and texture evolution in Cu layers of a roll-bonded Cu-Nb composite.* Materials Science and Engineering A, 2009. **520**(1-2): p. 189-196.

[2] Beyerlein, I.J., et al., *Interface-driven microstructure development and ultra high strength of bulk nanostructured Cu-Nb multilayers fabricated by severe plastic deformation.* Journal of Materials Research, 2013. **28**(13): p. 1799-1812.

[3] Zheng, S., et al., *Engineering Interface Structures and Thermal Stabilities via SPD Processing in Bulk Nanostructured Metals.* Scientific Reports, 2014. **4**: p. 4226.

[4] Zheng, S., et al., *High-strength and thermally stable bulk nanolayered composites due to twin-induced interfaces.* Nature Communications, 2013. **4**.

[4] “Tensile behavior of 40 nm Cu/Nb nanoscale multilayers”, N. Mara, D. Bhattacharyya, R.G. Hoagland and A. Misra, Scripta Materialia 58 (2008), 874-877

[5] “The effects of decreasing layer thickness on the high temperature mechanical behavior of Cu/Nb nanoscale multilayers”, N.A. Mara, T. Tamayo, A.V. Sergueeva, X. Zhang, A. Misra, A.K. Mukherjee, Thin Solid Films 515 (2007), 3241-3245

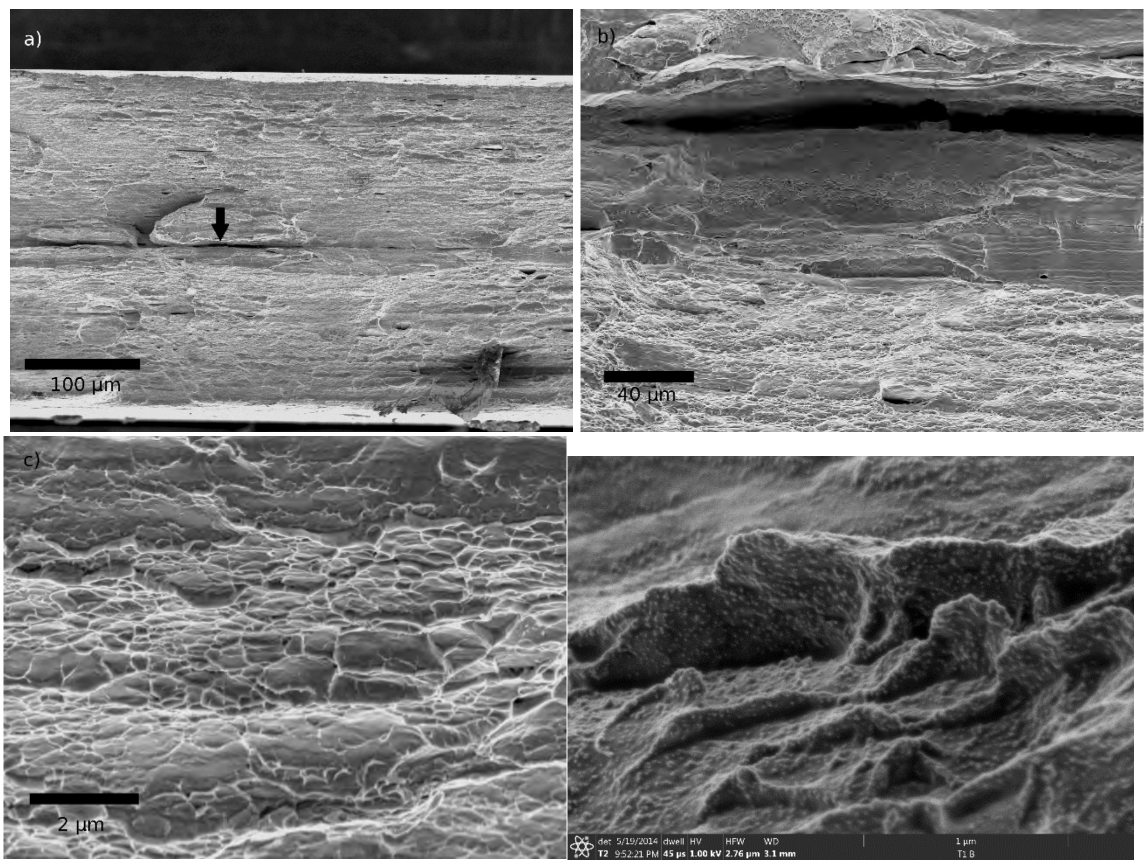

Fig. 3: a) Overview of the fracture surface tested at 20C. Tensile direction was parallel to the rolling direction. Closer look at the layer delamination in the center of the sample (marked by a black arrow in a). c) Typical structures on the fracture surface, d) at higher magnification (obtained with the Scios SEM from FEI)

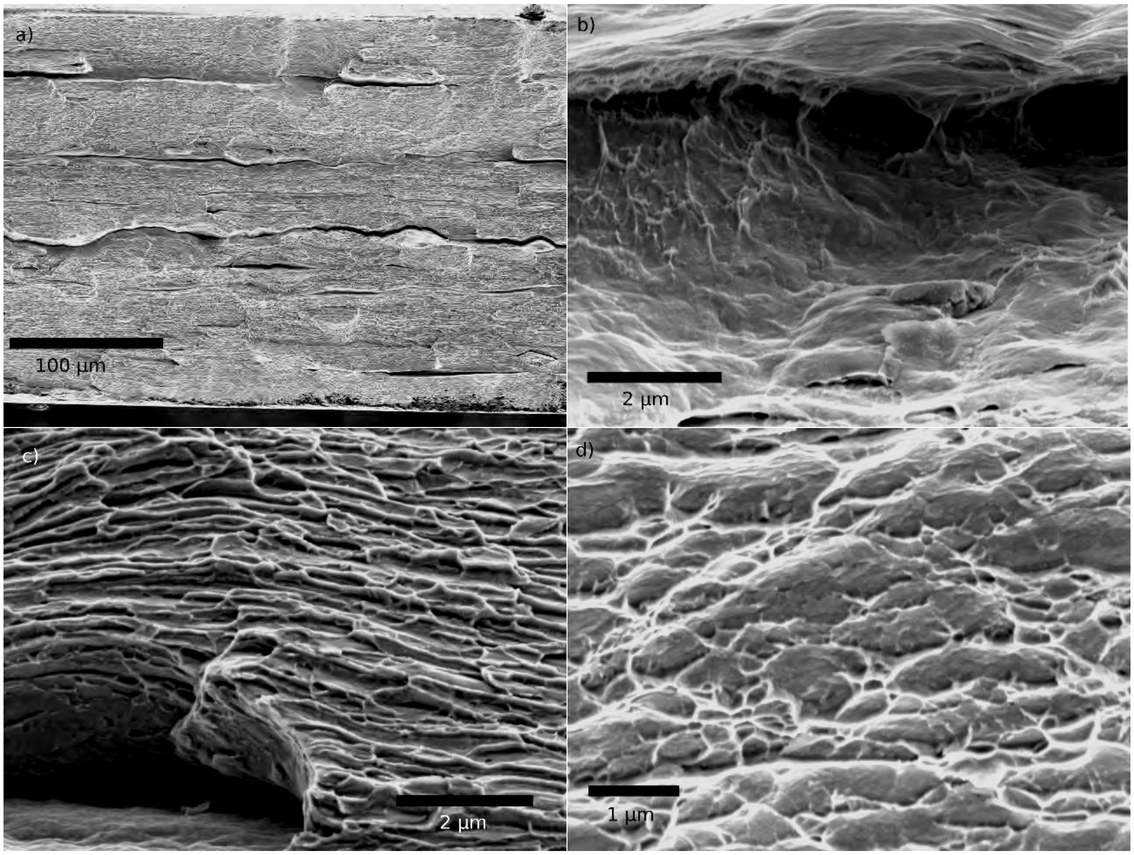

Fig. 4: a) Overview of the fracture surface tested at 500 C. Tensile direction was perpendicular to the rolling direction. Closer look at the layer delamination in the center of the sample (marked by a black arrow in a). c) and d) typical features found on the fracture surface